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Methods for Obtaining Calcium Nitrate

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Annotation: A detailed analysis of the physical and chemical properties of nitrite and sodium nitrate has been made, which must be taken into account when it is used in certain sectors of the national economy. An analysis of the literature data showed that the most acceptable method is the determination of sodium nitrite by titration. This method of analysis meets the requirements of expressiveness, accuracy, sensitivity, and the possibility of automating the control process under production conditions.

Key words: Nitrate, chemical, business.

The use of calcium nitrite as one of the components of dry mixes in construction is also relevant. Modern dry mixes used in construction are multicomponent specialized systems, which, in addition to the mineral binder and filler, contain a complex of chemical additives (in particular, sodium or calcium nitrite from 4 to 10% by weight of the dry mix), providing the necessary rheological properties of the mix, regulating the rate of setting and hardening of the binder and imparting the necessary physical and mechanical properties to the solution after hardening.

In the countries of Western Europe, the production and use of dry mixes in construction is massive. In terms of one inhabitant, the production of dry mixes in Germany is about 30 kg per year, and in Finland and Sweden about 20 kg; in Poland, where the licensed production of dry mixes has developed, this figure is about 23 kg [1].

The scientific literature provides a lot of data on the study of the physicochemical and structural properties of sodium nitrite.

The kinetics of the dissolution of cylindrical $NaNO_2$ particles in water is experimentally studied when they are located in a vertical tube through which the flow passes. The particles were obtained by casting in special molds and introduced into the flow on a long needle, the axis of which coincided with the axis of the tube. The recoil mass coefficient β was calculated from the change in particle weight. It is indicated that the results of the experiments are satisfactorily described by the equation:

$Id\ (I/d)^{-0,127} = 0,4Re^{-0,465}$ [2].

The paper presents the results of measurements of density, viscosity and surface tension of solutions of nitrite and sodium nitrate, as well as their mixed solutions, in the temperature range of 20-80 degrees Celsius up to a salt concentration of 40 wt%. The equations of connection of physical properties of solutions with salt concentration and temperature are given [3].

The study of the electrical conductivity of molten $NaNO_2$ at pressures up to 6 bar and heat capacity are given in [4]. Of theoretical interest are studies on the effect of ethyl alcohol on the growth rate of $NaNO_2$ crystals in aqueous solutions. The directional crystallization of sodium with an admixture of magnesium under the action of centrifugal forces was also studied.

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Phase transitions in NaNO2 were studied in [5]. In the studies, phase transitions were studied by the Monte Carlo method, which consists in calculating the Ising model with a dipole short-range potential on the $NaNO_2$ lattice. The cell consisted of 1024 dipoles, the dipole interaction with the rest of the macroscopic sample was considered based on a simplified version of the Ewald method. The temperature dependences of the heat capacity and spontaneous polarization show two maxima corresponding to phase transitions of the paraelectric. The characteristic configurations of dipoles (cell cross section) in three phases are established. The mathematical experiment is reproduced at the microscopic level.

Similar studies were carried out using the model of two sublattices of interacting dipoles. The molecular field approximation describes the phase transition from vapor to the ferroelectric state in $NaNO_2$ at 436 degrees Kelvin. The calculated polarization is in good agreement with the experimental data, however, in the antiferroelectric state between the temperatures TC and TN, the behavior of the substance under study is more complicated and cannot be described using the presented model.

The electron density distribution in $NaNO_2$ crystals was studied by X-ray diffraction [6]. The refinement at the beginning of the n-model of the crystal, and then the multipole model according to Hansen-Koppenz, was brought to R = 0.013, R = 0.019, S = 0.9. In the NO2 group, the N-O distance is 1.2574 A^0, and the ONO angle is 114.65^0, which made it possible to obtain much more pronounced features on the electron density maps. The authors attribute this to a more reliable determination of phases upon reflection in a noncentrosymmetric $NaNO_2$ crystal using this model. At the same time, similar results were obtained by refining early experimental data for centrosymmetric $LiNO_2{\cdot}H_2O$. The electron density maps showed that Na atoms are characterized by the state of sp^2 hybridization. Electronic obscurations δ - orbitals, determined using the model of oriented atoms, had values greater than unity.

The influence of exciton dispersion on the line shape of the fluorescence spectrum of $NaNO_2$ is given in the work.

The vibrational structure of $NaNO_2$ fluorescence consists of asymmetric lines, which is due to the exciton dispersion of the excited state and the vibrational dispersion of the ground state. The excitation spectrum and its dependence on the detected energy and temperature are studied. The nature of the dependence on the detected energy rarely changes at a critical temperature of about 15^0K. This allows us to conclude that, at low temperatures, excitons are not terrorized during their lifetime.

When developing a technology for the production of sodium nitrite, a very important issue is the correct choice of the method of analysis. The literature describes various methods for the analysis of sodium nitrate and nitrite, depending on the object of analysis. A number of analytical methods relate to the determination of sodium nitrite in meat and meat products.

The paper attempts to conduct a comparative assessment of the determination of nitrates and nitrites in meat and meat products. The most accurate of the existing ones is the method using $FeCl_3$. The xylenol method is inaccurate and cumbersome. The method of direct calorimetry for the determination of nitrates, based on the nitration of aromatic compounds, is sensitive and accurate, but requires the use of concentrated sulfuric acid.

Landmann methods with brucine and West, Lyles method with chromotropic acid can be used. The results of these methods are in good agreement with the data of the method with $FeCl_3$. Other determination methods based on the reduction of nitrates to ammonium and spectrophotometry are not applicable to meat products.

According to some methods, nitrates are reduced to nitrites and determined in total; nitrates are calculated by the difference before and after recovery; these methods are fast and accurate. For nitrate-nitrite determination, the iodometric method People is fast and accurate. It gives good results in the determination of nitrite in brines. The determination method with phenolphthalein gives satisfactory results.

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A fast method for determining the content of nitrites in meat products is proposed, based on the formation of a pink color by solutions of sulfanilic acid and α - naphthylamine in acetic acid in the presence of nitrites. The color intensity depends on the nitrite content. When comparing the color intensity of the test solution with a model solution (for example, 15 mg/100 g $NaNO_2$) for 30 minutes, it can be established whether the nitrite content in the test product is below or above the limit established by the standard.

The methods propose a modified Griess method for determining the content of nitrites in meat and meat products, which consists in replacing α - naphthylamine with phenol, which makes it possible to eliminate the inconveniences of this method, namely: to achieve color stability, apply the Lambert-Beer law, increase sensitivity and speed analysis. The determination error is less than 1%.

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A quick method for determining the content of nitrites in meat products is proposed, based on the formation of a pink color by solutions of sulfanilic acid and -naphthylamine in acetic acid in the presence of nitrites. The color intensity depends on the nitrite content. When comparing the color intensity of the test solution with a model solution (for example, 15 mg/100 g $NaNO_2$) for 30 minutes, it can be established whether the nitrite content in the test product is below or above the limit established by the standard.

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Japanese scientists have developed a method for analyzing nitrites in wastewater from dye factories. To determine the concentration of NO_2 (in the range of 1:10 mg/l) in colored wastewater, the latter is passed through a column filled with Dowex anion exchanger in OH-form. wastewater pH is maintained in the range of 5-9; flow rate through the column $\approx$ 7-10 ml/min. Adsorbed NO_2^- ions are washed out with 0.1 n HC1 and their concentration in the eluate is determined by the calorimetric method.

A spectrophotometric method for the determination of $NaNO_2$ in mixtures based on differences in absorption at 354 and 302 nm is proposed. $NaNO_2$ is absorbed at 354 nm, where there is no $NaNO_3$ absorption, and can be detected at this band. At 302 nm both salts are absorbed, but the absorbance of $NaNO_3$ can be obtained from the absorbance of 302 nm by subtracting the absorbance of the 354 nm band from it divided by 2.57.

The analysis of mixtures of $NaNO_2$ and $NaNO_3$ must meet the requirements of rapidity, accuracy, sensitivity of analysis, and the possibility of automating the control process under production conditions.

Among the methods described in the literature for the determination of nitrates and nitrites in the joint presence, the simplest to perform and meet the above requirements are the methods of redox titration. The use of the Lunge method for determining the content of sodium nitrite in salt solutions of the following composition: $NaNO_3$ - $NaNO_2$ - Na_2CO_3 - $NaHCO_3$ - NaC1 - H_2O gives accurate and well reproducible results.

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As a result of experimental studies, the authors concluded that for technological solutions for the production of sodium nitrite and sodium nitrate, the most appropriate method for determining nitrite ions is by titration with a standard solution of ascorbic acid in the presence of phosphoric acid, magnesium sulfate and ferroin.

Ascorbic acid reduces NO to nitric oxide, Mn^{2+} ions catalyze the reduction process, provided that the titrated solution is 10 M in H_3PO_4. The ferroin indicator changes color from blue to orange at the equivalence point. At a concentration of H_3PO_4 in a solution of 10 M, the catalytic effect of Mn^{2+} does not manifest itself, and when the first drop of an ascorbic acid solution is introduced, the indicator changes its color irreversibly.

Combining the two above-mentioned methods for determining $NaNO_2$ and NaNO3 made it possible to find the content of nitrite and nitrate in one sample.

The essence of the method for determining nitrite and nitrate is as follows. A hot acidified solution of potassium permanganate is titrated from a burette with an analyzed solution containing $NaNO_2$ and $NaNO_3$; after titration, a solution is found in the flask, where the nitrate contained earlier in the solution is present, and the one that was formed due to the oxidation of nitrite. The total content of nitrate in this solution is determined by titrating the solution in the presence of H_3PO_4 and ferroin with a standard solution of ascorbic acid. In this case, there is no need to specially add a manganese (II) salt to the titrated solution, since Mn^{2+} ions are formed during the reduction of permanganate.

A device for the determination of nitrates in natural and waste waters has been developed [7]. The operation of the device is based on their reduction to nitrites, followed by determination by the Griess-Ilosval reaction. A disk rotating at a given speed brings cups with devices with distilled water in series to the suction inlet of the pump, the latter takes 2 ml of a sample and brings it to 2 dialyzers placed in series. A buffer acetate solution is pumped into the sample after the dialyzers, after which it is pumped through a column with granular Zn, where NO_3- is reduced to NO_2-. Next, a reagent is added to the sample, composed of equal volumes of solutions of sulfanilic acid and naphthylamine. After passing through the mixing coil, the buffer solution is again added to the sample, the liquid is mixed in the coil and heated to 50°C. The colored solution flows continuously through the calorimeter cell (10 mm), light absorption is measured at λ 520 мμ and marked by a chart recorder on a moving map. After taking each sample, the device is washed with distilled water, taken by a pump from cups placed next to the sample. At a concentration of $N_{nitrate}$ of 0.05–10 mg/l, the standard deviation from the mean is 1.3–6%.

In recent years, the method of analysis of technical sodium nitrite by titration has become generally accepted. According to this method, solutions of sulfuric acid, potassium permanganate, potassium iodide are added to an aqueous solution of $NaNO_2$, mixed and titrated with a solution of sodium sulphate to a slightly yellow color, after which a starch solution is added and titration is continued until the solution becomes colorless.

Such close attention of researchers to the properties and methods of analysis of nitrate nitrite compounds is explained by their ever more widespread use in various industries.

The properties, production and use of calcium nitrate in agriculture are described in detail in the monograph by Beglov B.M. and others. The need for calcium for plants was already recognized by Saussure (1804) on the basis of an analysis of the ashes of plants, which always contain calcium.

Studies have confirmed that calcium has a significant effect on the metabolism of both carbohydrates and nitrogenous substances in plants. In the experiments of N.S. Turkova, a sharp change in the composition of various groups of nitrogenous substances in wheat germs under the influence of a change in the ratio between calcium and potassium in the nutrient mixture was stated. An increase in the calcium concentration

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in the solution was accompanied by an increase in the content of protein and amide nitrogen and a decrease in the nitrogen content.

For the development of a normal root system, the need for the presence of calcium ions in the solution surrounding the roots is most clearly revealed; undesirable changes in cell walls occur if calcium is not present in the surrounding solution or if it is displaced from compounds (with lipoids, pectin substances, etc.) by a one-sided excess of Mg, K or Na.

In addition to monovalent and divalent cations (H, K, Na, Mg), calcium can also neutralize ions of trivalent metals (AI and Fe), which may be present in an acidic environment (in particular, in acidic soils).

Calcium ions have a specific effect on the properties of monomolecular films of cell membranes and the stability of these formations depends on the presence of calcium. Influencing the permeability of membranes, it regulates the exchange of ions between plant cells and the external environment.

Calcium is necessary to maintain the structure of the mitochondria of cells; in the absence of it, the mitochondria are destroyed. Calcium ions are specific activators of phosphatase, lecithinase, lipase and apyrase. In addition, together with other ions, they are involved in the activation of pantetheine phosphokinase, aldehyde dehydrogenase, oxaloacetate decarboxylase, α-carboxylase, etc. At the same time, it inhibits pyruvate phosphokinase, ATPase, nucleotidase, flavokinase, and a number of other enzymes involved in the oxidation of amino acids.

Consequently, the role of calcium in the metabolism of various substances in plant tissues is enormous. Therefore, it belongs to the macronutrients in which the plant has a significant need.

Calcium is a widespread element: its content in the earth's crust is 3.25%. It is present in the soil in the form of silicates, carbonates, gypsum, in the soil solution - in the form of salts: nitrate, bicarbonate, chloride, etc. Calcium has a great influence on soil fertility.

The main source of plant nutrition with calcium is the calcium of the soil solution, absorbed and carbonate calcium.

The lack of calcium may be due to its loss from the soil due to leaching and as a result of the removal of this element with crop yields. The removal of calcium by a crop of grain crops is 20-40 kg, peas and flax - 43-60 kg, sugar beet - 70-80 kg, potatoes - 10 kg, alfalfa and clover - 100-250 kg, grass (hay) - 70 kg, cabbage 300-400 kg CaO per hectare. Naturally, these calcium losses increase with crop yields. However, in practice, the loss of calcium during soil leaching and the use of fertilizers that increase soil acidity (ammonium nitrate) is much greater than it is taken out with the crops.

For cotton on sandy and sandy loamy soils, the minimum content of calcium soluble in an acetate buffer solution was set to be approximately 200 mg/kg of soil, and for calcium soluble in 0.5 N HCI-300 mg/kg, for light loamy and silty loamy soils it was equal to respectively 200 and 500 mg/kg.

Therefore, permanent liming is a necessary procedure on all soils that do not have natural sources of lime. Even in areas with low rainfall, the application of lime should be at least 3-4 t CaO per 1 ha or 5-6 t $CaCO_3$ (crushed) per 1 ha. These measures need to be repeated every five years to replace calcium losses on heavily used agricultural land due to leaching and the application of fertilizers rich in nitrogen and potassium.

For acidic soils, liming is a measure to combat the disease, for the rest - the introduction of calcium nitrate, calcium chloride, gypsum, phosphogypsum, simple superphosphate, which, however, does not completely eliminate calcium starvation. A more effective measure is spraying plants during the period of strong fruit growth with a 0.5-1% solution of calcium nitrate or calcium chloride (1-2 times a week). The most

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promising results are obtained by the combination of these two methods: it not only suppresses the disease, but also increases the yield [8].

Calcium nitrate is widely distributed in nature, in all cultivated soils and in most soils under wild vegetation; The nitric acid formed during the nitrification process binds to calcium, so the nitrogen supply to plants occurs mainly from $Ca(NO_3)_2$.

Thus, the best form for introducing nitrogen into soils with insufficient calcium content, since in this case, unlike sodium nitrate and ammonium salts, even with abundant and frequent application, not only can there not be a deteriorating effect on the soil structure, but vice versa, on In podzolized soils, a gradual improvement in physical properties should occur due to the incorporation of calcium into the absorbing complex instead of hydrogen.

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